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Michael Mooney

Michael Mooney
APDS, AEGD-2 Residency
Fort Hood, TX
Uniformed Services University
Date: 05/01/2016

Uniformed Services University
of the Health Sciences

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INITIATOR

1. USU Principal Author/Presenter: Mooney, Michael CPT, DC

2. Academic Title: Dr/Senior Resident

3. School/Department/Center: Army Postgraduate Dental School, AEGD-2 Program, Fort Hood, TX

4. Phone: (678) 373-7629

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CHAIR OR DEPARTMENT HEAD APPROVAL

1. Name: Mark McClary, COL, DC, Director

2. School/Dept.: Army Postgraduate Dental School, AEGD-2 Program, Fort Hood, TX

3. Date: 1 May 2016

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Chair/Department Head Approval: Mark McClary Date_1 May 2016_

COMMANDER APPROVAL

1. Name: Michael E. Garvin, COL, DC, Commander

2. U.S. Army Dental Activity, Fort Hood, Texas

3. Date: 2 May 2016

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1. Name: Douglas D. Lancaster, COL, DC, Dean

2. School: Army Postgraduate Dental School

3. Date:

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Service Dean Approval: LANCASTER.DOUGLAS.DUNN.1120129313 Date________

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2. School: Postgraduate Dental College

3. Date:

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COMPARATIVE Shear Bond Strength of Resin Buttons to Lithium Disilicate and Leucite Reinforced Feldspathic Restorations

A Thesis
Presented to the Faculty of the Advanced Education in General Dentistry, Two-Year Program,
United States Army Dental Activity, Fort Hood, Texas
And the Uniformed Services University of the Health Sciences – Post Graduate Dental College
In Partial Fulfillment of the Requirements for the Degree of
Master of Science in Oral Biology

By

MICHAEL A MOONEY, CPT, USA, DC

April 2016

COMPARATIVE Shear Bond Strength of Resin Buttons to Lithium Disilicate and Leucite Reinforced Feldspathic Restorations

A REPORT ON
Research project investigating the Shear Bond Strength of Resin Buttons to Lithium Disilicate and Leucite Reinforced Feldspathic Restorations

By
MICHAEL A MOONEY, CPT, DC, USA
D.M.D., Tufts School of Dental Medicine, 2013
B.S., Emory University, 2009

Mentor Staffing By
Mark Church, MAJ, DC, USA
D.M.D., University of Kentucky College of Dentistry, 2008

Fort Hood, Texas
April 2016

ABSTRACT

Purpose: The purpose of this study is to compare the shear bond strength of resin buttons to lithium disilicate (IPS e.max® CAD) and leucite reinforced feldspathic restorations (Empress CAD) after different surface preparation techniques.

Methods and Materials: Group A,B,C,D, and E are divided by the surface treatments that were applied. IPS e.max® CAD crowns were fired in porcelain oven according to manufacturer recommendations. One sample of each group was photographed with scanning electron microscopy (SEM).

1) Group A: silane coupling agent, light cured composite, and ceramic restoration specimens (10 of each IPS e.max and Empress) in this group had a silane coupling agent applied and air dried twice (Scotchbond Universal). A standard composite button (Filtek Supreme Ultra) was applied with a button former and light cured for 20 seconds.

2) Group B: 9.6% Hydrofluoric acid, silane coupling agent, light cured composite and ceramic restoration specimens (10 of each IPS e.max and Empress). The surface treatment in group B was applied to all specimens: Ceramic (IPS e.max and Empress) specimen surfaces were etched with 9.6% hydrofluoric acid gel (Pulpdent) for 20 seconds and rinsed. All specimens were washed, rinsed and air dried to remove any residual acid etchant. A silane coupling agent was used to condition the specimen surfaces (Scotchbond Universal) and was applied with a microbrush and air dried twice. A standard composite button (Filtek Supreme Ultra) was applied with a button former and light cured for 20 seconds.

3) Group C: 9.6% Hydrofluoric acid, silane coupling agent, light cured composite and ceramic restoration specimens (10 of each IPS e.max and Empress). The surface treatment in group C was applied to all specimens: Ceramic (IPS e.max and Empress) specimen surfaces were etched with 9.6% hydrofluoric acid gel (Pulpdent) for 60 seconds and rinsed. All specimens were washed, rinsed and air dried to remove any residual acid etchant. A silane coupling agent was used to condition the specimen surfaces (Scotchbond Universal) and was applied with a microbrush and air dried twice. A standard composite button (Filtek Supreme Ultra) was applied with a button former and light cured for 20 seconds.

4) Group D: 5% Hydrofluoric acid, silane coupling agent, light cured composite and ceramic restoration (10 of each Empress and IPS e.max). The surface treatment in group D was applied to all specimens: IPS e.max and Empress ceramic restorations were etched for 20 seconds with 5% hydrofluoric acid gel. All specimens were washed, rinsed and air dried to remove any residual acid etchant. A silane coupling agent was used to condition the specimen surfaces (Scotchbond Universal) and was applied with a microbrush for 20 seconds and air dried twice. A standard composite button (Filtek Supreme Ultra) was applied with a button former and light cured for 20 seconds.

5) Group E: 5% Hydrofluoric acid, silane coupling agent, light cured composite and ceramic restoration (10 of each Empress and IPS e.max). The surface treatment in group E was applied to all specimens: IPS e.max and Empress ceramic restorations were etched for 60 seconds with 5%

hydrofluoric acid gel. All specimens were washed, rinsed and air dried to remove any residual acid etchant. A silane coupling agent was used to condition the specimen surfaces (Scotchbond Universal) and was applied with a microbrush for 20 seconds and air dried twice. A standard composite button (Filtek Supreme Ultra) was applied with a button former and light cured for 20 seconds.

Specimens were stored for 24hrs in distilled water and then loaded into a universal testing machine (Instron) for testing, with the long axis of the specimen perpendicular to the direction of the applied force. A notch edge shear blade was positioned to make contact with the bonded specimen. Bond strength was determined in shear mode at a crosshead speed of 0.75 +/- 0.3 mm/minute until fracture occurred.

Results: IPS e.max® CAD displayed statistically significant higher shear bond strength at 19.29 MPa when hydrofluoric 9.6% was applied for 20 seconds and hydrofluoric 5% applied for 60 seconds at 18.59 MPa when compared to the other test groups.

Conclusions: IPS e.max® CAD etched with 9.6% hydrofluoric acid for 20 seconds showed the highest shear bond strength of all materials tested with IPS e.max® CAD etched with hydrofluoric 5% acid for 60 seconds also statistically significant. Within the limitations of this study, the results can help to determine an etch and bond protocol for bonding composite to IPS e.max® CAD or Empress CAD.

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TABLE OF CONTENTS

INTRODUCTION

Orthodontics has been intimately linked with dentistry for more than 2000 years.[1] The origin of dentistry comes from a part of medicine. According to the American Association of Orthodontists (AAO), archaeologists have discovered mummified remains with metal bands wrapped around individual teeth.[1] Malocclusion is not classified as a disease, but an abnormal alignment of the teeth and the way in which the upper and lower teeth fit together.[3] The prevalence of malocclusion varies, but it can be extrapolated that nearly 30% of the population presents with malocclusions severe enough to benefit from orthodontic treatment.[2,3,4]

Orthodontists traditionally used metal braces with wires to align and straighten teeth and help to position them into the correct occlusion to improve function, esthetics, and dental health. Metal braces are fixed orthodontic appliances that are bonded to the teeth utilizing enamel bonding procedures. These fixed orthodontic appliances are the gold standard in orthodontic treatment and can be used to correct many types of malocclusions to include: underbites, overbites, cross bites, open bites, deep bites, mal-aligned teeth, and used in conjunction with orthognathic surgery. Dental braces can be used with other fixed or removable orthodontic appliances to help widen the palate or jaws and to otherwise assist in shaping the teeth and jaws. Before the mid-1990s, only minor tooth movement and interceptive orthodontic treatment was commonly done in the United States with removable dental appliances.

Invisalign was created by Align Technologies in 1997. Invisalign is a method of orthodontic treatment which uses a series of clear, removable teeth aligners used as an alternative to traditional metal dental braces. Manufacturer statistics show 730,000 patients have completed or are currently in treatment. Since the advent of Invisalign, many orthodontists and general practitioners are now trained in clear aligner therapy to treat dental malocclusions. Certain movements with clear aligner therapy require the bonding of composite resin buttons to apply a more consistent force to the tooth. This allows for more predictable tooth movements. When either traditional orthodontic brackets or resin buttons need to be bonded to natural teeth standard protocol is followed to achieve a predictable bond of resin material to the enamel of teeth. When this same procedure needs to be done on a restorative material such as lithium disilicate, or leucite reinforced feldspathic porcelain it can be a challenging task.

Dentists are now regularly using clear aligner therapy to correct malocclusions. Patients who do not want to have traditional band and bracket orthodontics are now requesting treatment with clear aligner therapy. Some individuals who seek orthodontic care with clear aligners have all porcelain restorations and need resin buttons bonded to the restoration in order to achieve proper tooth movement. Protocols have been proposed for porcelain repair with composite resin and resin bonding of ceramic restorations to teeth. Bonding composite to porcelain is not very predictable. This bench top pilot study was conducted to test bond strength for bonding resin buttons to porcelain with different surface treatments. The results of this study will provide a protocol for surface treatment of porcelain to bond composite buttons or perform composite repair of porcelain crowns.

The first material tested is IPS e.max CAD which is composed of lithium disilicate glass-ceramic and zirconium oxide. IPS e.max presents with two forms for dental use: a homogeneous ingot with various degrees of opacity used with hot-pressed technology through the lost wax technique and a pre-crystallized block used with CADCAM technology. Both forms can be used in a full anatomical contour method with the application of stain and glaze or a cutback and layering technique.

Glass technology is used to produce the CAD milling blocks which prevents defects and voids. [5] The glass technology also allows the pigmentations added to the block for shade selection to be evenly distributed. [5] IPS e.max employs a partial crystallization process that forms the lithium-metasilicate crystals, which allow more efficient milling while adding good edge stability properties to prevent chipping while milling.[5] Partially crystallized IPS e.max milling blocks consists of 40 percent lithium-metasilicate crystals embedded in a glassy matrix with the grain size of these crystals ranging from 0.2 µm to 1 µm.[5] While partially crystallized in the purple state, the lithium metasilicate block has a flexural strength of 130-150 MPa.[5] The partially crystallized IPS e.max block is milled and can be placed in the mouth and adjusted if needed. The restoration is then placed in the oven at 850 degrees Celsius to be fully crystallized. The size difference between partially crystallized IPS e.max and fully crystallized IPS e.max is 0.2 percent which the computer software calculates and adjusts for during the milling process. The lithium metasilicate crystals grow and transform to lithium disilicate during the temperature rise increasing the final flexural strength to 360 MPa.[5] The finalized product, lithium disilicate, is comprised of 70 percent prismatic lithium disilicate crystals (0.5 µm to 5 µm long)(Fig. 1) interspersed in a glassy matrix.[5] Lithium disilicate gains its additional flexural strength and fracture toughness from its crystal size and orientation which causes cracks to deflect instead of propagate.

The second material being tested, IPS Empress CAD, is a leucite glass-ceramic in a glassy matrix.[6] The leucite crystals are formed in a controlled process endowing the material with an increased strength.[6] The leucite crystals increase the strength of Empress by slowing down or deflecting the propagation of cracks.[6] The fracture energy is absorbed by the crystalline phase resulting in the deceleration or arrest of the cracks.[6] The leucite crystals of Empress are formed by surface crystallization in which the crystals grow slowly beside the grain boundaries continuing towards the center of the grain.[6] The diameter of the crystals is 1 – 5 µm (Figure 2). The leucite crystals of Empress CAD give the material a flexural strength of 160 Mpa.[6]

Two different concentrations of hydrofluoric acid are tested in this study to determine which provides the best shear bond strength. These two strong hydrofluoric acids create a series of surface pits by preferential dissolution of the glass phase from the ceramic matrix.[7] This process microscopically roughens the porcelain to enhance micromechanical retention of composite resin to the porcelain.[8] With hydrofluoric acid care must be exercised due to its extremely corrosive nature which can cause severe trauma to soft tissues and tooth substance. [9] Manufacturer recommendations for IPS e.max recommend 5% hydrofluoric acid for 20 seconds when etching to deliver single unit crowns. Manufacturer recommendations for Empress recommend 9.6% hydrofluoric acid for 60 seconds when etching to deliver single unit crowns.

Bernas et. al looked at the shear bond strengths of self-adhering resin to different restorative materials and found that self-adhesive resins could provide clinically acceptable bond strengths to enamel, restorative resin composite and porcelain with minimal surface preparation.[10] The bonding of orthodontic brackets to restorative materials has been studied by many including Bayram, Bilgic, Bishara, and Trakyalı. These studies came to the consensus that orthodontic brackets bonded to ceramic substrate should include a mechanical or micromechanical roughening of the ceramic surface, through direct abrasion from rotary instrumentation or air abrasion.[10,11,14,15,16] The bonding should also include acid etching with hydrofluoric acid and silanation to increase bond strength of the resin cement to the restorative material. Panah, Kim, Bourke, Eustaquio, and Girish looked at the surface treatment of all ceramic materials and the bond strength based on surface preparation showing silane coating after air abrasion and etching improved the bond strength.[12,13,17,18,19]

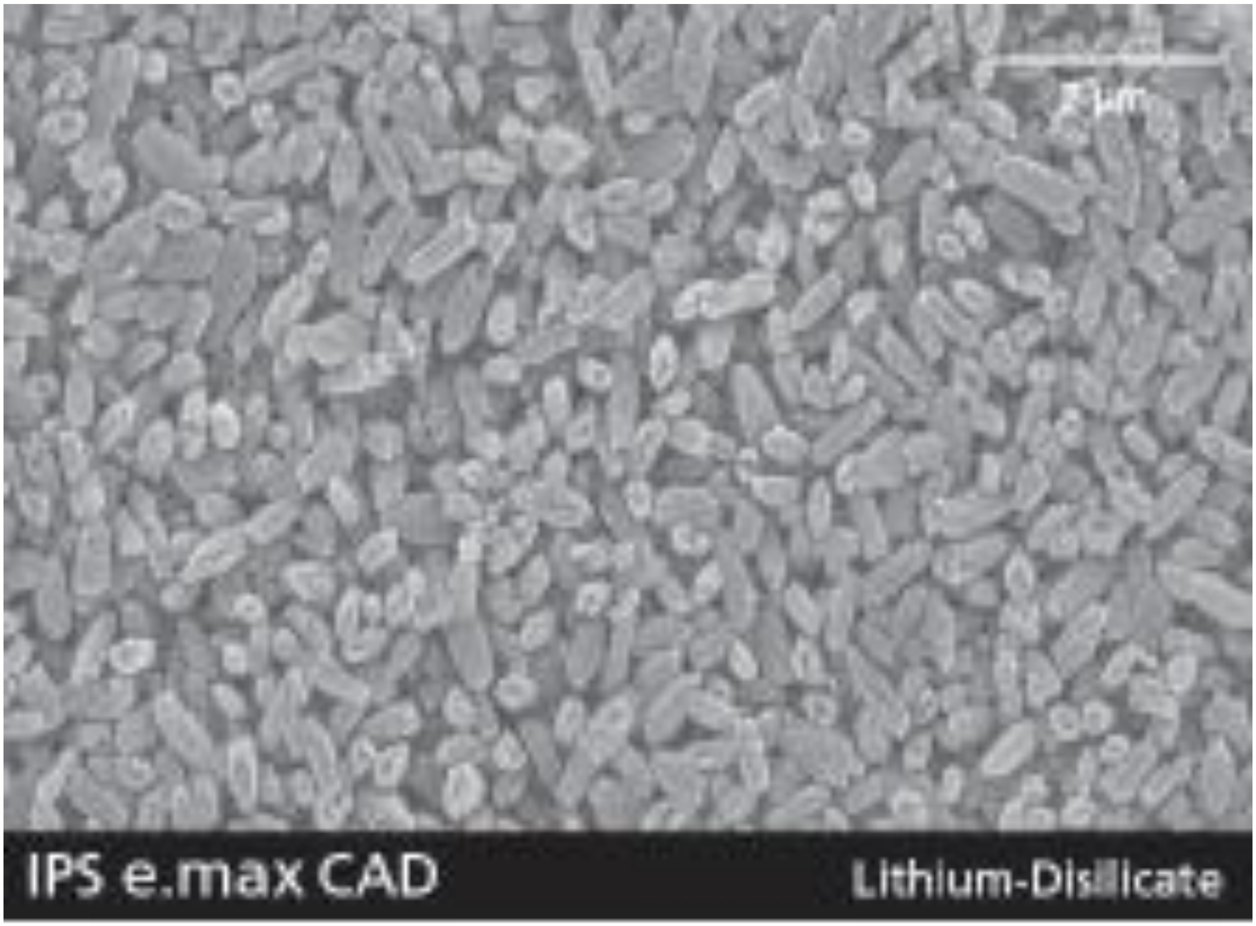


Figure 1. IPS e.max® CAD crystallized (avg. size 1.5 µm)

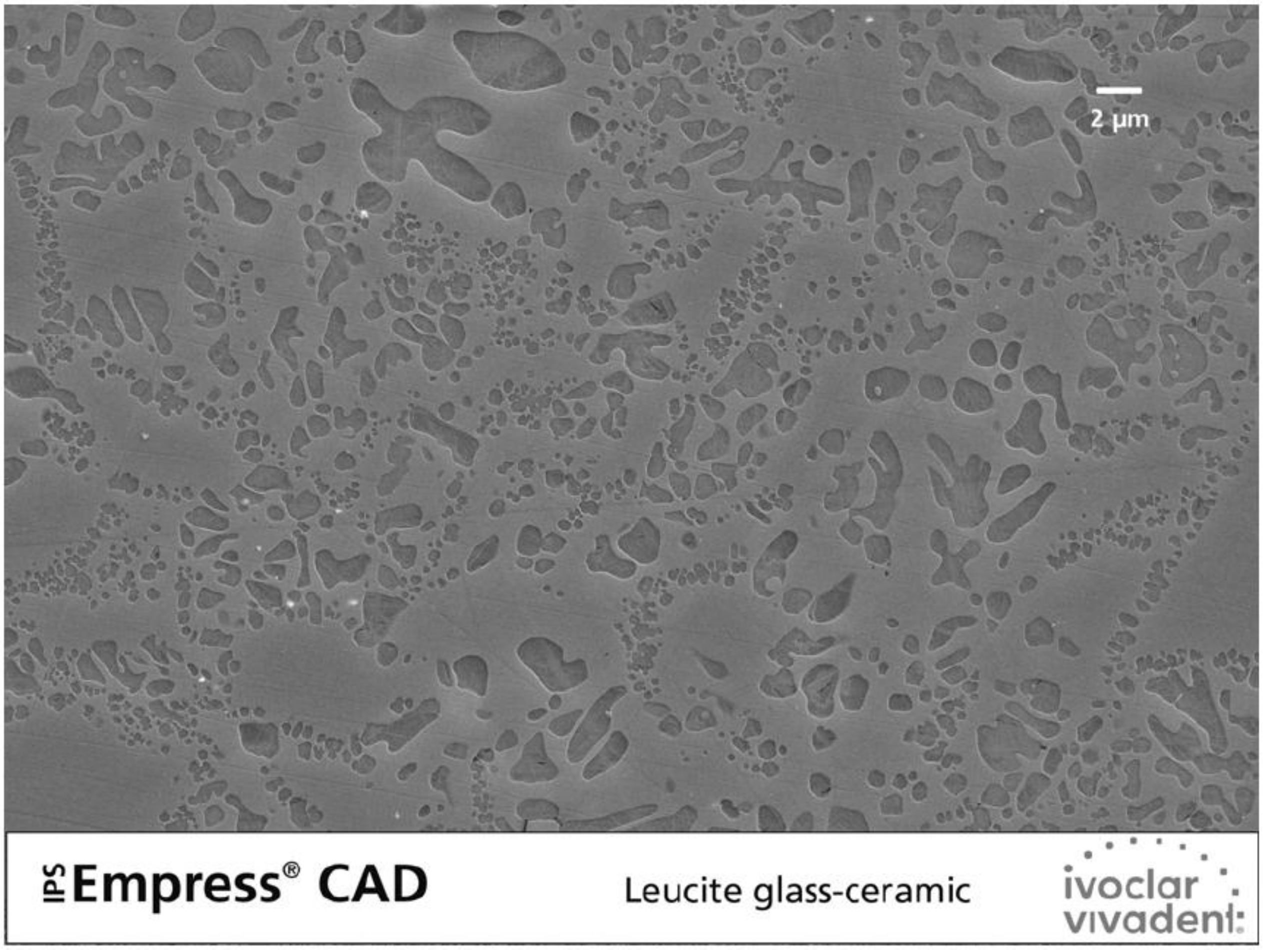


Figure 2. Empress™ CAD (1-5 µm)

PURPOSE

The purpose of this study is to compare the shear bond strength of resin buttons to lithium disilicate (IPS e.max® CAD) and leucite reinforced feldspathic restorations (Empress CAD) after different surface preparation techniques.

HYPOTHESES

Research question: Will there be a difference in shear bond strength when comparing different surface preparations on IPS e.max® CAD and Empress CAD to bond composite buttons?

Null hypothesis: There will be no difference in shear bond strength between lithium disilicate and leucite reinforced feldspathic restorations regardless of the surface preparation used.

METHODS AND MATERIALS

Group A,B,C,D, and E are divided by the surface treatments that are applied. IPS e.max® CAD crowns were fired in porcelain oven according to manufacturer recommendations.

Group A consisted of a silane coupling agent and light cured composite. The ceramic restoration specimens (10 of each IPS e.max and Empress) in this group had a silane coupling agent applied to condition the specimen surfaces (Scotchbond Universal) with a microbrush and air dried twice. A standard composite button (Filtek Supreme Ultra) that was applied with a button former and light cured for 20 seconds.

Group B consisted of 9.6% hydrofluoric acid, silane coupling agent, light cured composite and ceramic restoration specimens (10 of each IPS e.max and Empress). The surface treatment in group B was applied to all specimens. Ceramic (IPS e.max and Empress) specimen surfaces were etched with 9.6% hydrofluoric acid gel (Pulpdent) for 20 seconds and rinsed. All specimens were washed, rinsed and air dried to remove any residual acid etchant. A silane coupling agent was used to condition the specimen surfaces (Scotchbond Universal) and was applied with a microbrush and air dried twice. A standard composite button (Filtek Supreme Ultra) was applied with a button former and light cured for 20 seconds.

Group C consisted of 9.6% hydrofluoric acid, silane coupling agent, light cured composite and ceramic restoration specimens (10 of each IPS e.max and Empress). The surface treatment in group C was applied to all specimens. Ceramic (IPS e.max and Empress) specimen surfaces was etched with 9.6% hydrofluoric acid gel (Pulpdent) for 60 seconds and rinsed. All specimens were washed, rinsed and air dried to remove any residual acid etchant. A silane coupling agent was used to condition the specimen surfaces (Scotchbond Universal) and was applied with a microbrush and air dried twice. A standard composite button (Filtek Supreme Ultra) was applied with a button former and light cured for 20 seconds.

Group D consisted of 5% hydrofluoric acid, silane coupling agent, light cured composite and ceramic restoration (10 of each Empress and IPS e.max). The surface treatment in group D was applied to all specimens. IPS e.max and Empress ceramic restorations was etched for 20 seconds with 5% hydrofluoric acid gel. All specimens were washed, rinsed and air dried to remove any residual acid etchant. A silane coupling agent was used to condition the specimen surfaces (Scotchbond Universal) and was applied with a microbrush for 20 seconds and air dried twice. A standard composite button (Filtek Supreme Ultra) was applied with a button former and light cured for 20 seconds.

Group E consisted of 5% hydrofluoric acid, silane coupling agent, light cured composite and ceramic restoration (10 of each Empress and IPS e.max). The surface treatment in group E was applied to all specimens. IPS e.max and Empress ceramic restorations was etched for 60 seconds with 5% hydrofluoric acid gel. All specimens were washed, rinsed and air dried to remove any residual acid etchant. A silane coupling agent was used to condition the specimen surfaces (Scotchbond Universal) and was applied with a microbrush for 20 seconds and air dried twice. A standard composite button (Filtek Supreme Ultra) was applied with a button former and light cured for 20 seconds.

Specimens were stored for 24hrs in distilled water and then loaded into a universal testing machine (Instron) for testing, with the long axis of the specimen perpendicular to the direction of the applied force. A notch edge shear blade was positioned to make contact with the bonded specimen. Bond strength was determined in shear mode at a crosshead speed of 0.75 +/- 0.3 mm/minute until fracture occurred.

Data analysis. In this study, the independent variables are ten combinations of material and treatments. The dependent variable is shear bond strength. The null hypothesis is that there is no difference in the shear bond strength between combinations. The alternative hypothesis is that there is a difference in the shear bond strength between combinations. The appropriate statistical test is a one-way ANOVA followed by independent sample t-tests corrected for multiple comparisons. If the dependent variable is not normally distributed with equal variance, the equivalent non-parametric tests was used.

Sample size estimation/power analysis. A general analysis was performed. We used the on line power analysis program at the University of British Columbia (www.stat.ubc.ca/~rollin/stats/ssize/n2.html) to estimate the sample size needed for a power of 80% with a level of confidence of 95%. Seven comparisons among 10 combinations (groups) are appropriate for this study, so we used a Bonferroni correction of $p = 0.05 / 7 = 0.007$. With 101 samples per group (1010 total), the investigator was able to detect a moderate 0.5 SD effect size. With 40 samples per group (400 total), the investigator was able to detect a large 0.8 SD effect size. With 26 samples per group (260 total), the investigator was able to detect a larger 1.0 SD effect size.

The investigator plans to use 10 samples per group. With 10 samples per group (100 total), the investigator was able to detect a 1.5 SD effect size. If non-parametric tests are used the sample size should be increased by 15%.

RESULTS

The results of each group with surface preparations are listed in Tables 2-11. Table 12 and 13 display the difference between the shear bond strengths averages of the groups with IPS e.max prepared with 9.6% hydrofluoric for 20 seconds achieving the highest average shear bond strength at 19.29 MPa (p=<.0001).

Using the Tukey-Kramer for comparison shows a statistically significant difference in IPS e.max prepared with 9.6%hydrofluoric for 20 seconds and 5% hydrofluoric for 60 seconds (p=.01). Table 14 displays the connecting letters report and difference between shear bond strength of surface preparation.

For bonding composite buttons to the samples IPS e.max provided almost an average of 5 MPa more than Empress as Table 14 shows while the control of Scotchbond only has a 2 MPa difference in strength.

TABLES

Table 1. Groups with surface preparations

Group	Material	Preparation	Etching	Samples
1	IPS e.max	None	None	10
2		5% hydrofluoric	20 seconds	10
3			60 seconds	10
4		9.6% hydrofluoric	20 seconds	10
5			60 seconds	10
6	Empress	None	None	10
7		5% hydrofluoric	20 seconds	10
8			60 seconds	10
9		9.6% hydrofluoric	20 seconds	10
10			60 seconds	10

Table 2. Group 1- IPS e.max with Scotchbond

Sample	Diameter	Radius	Surface Area	Peak Load (N)	Peak Stress (MPa)
1	2.37	1.185	4.411502944	45.683	10.35542775
3	2.37	1.185	4.411502944	84.836	19.23063434
4	2.37	1.185	4.411502944	61.154	13.86239583
5	2.37	1.185	4.411502944	51.226	11.61191563
6	2.37	1.185	4.411502944	40.656	9.215906805
7	2.37	1.185	4.411502944	18.422	4.175901101
8	2.37	1.185	4.411502944	21.235	4.813552268
9	2.37	1.185	4.411502944	65.366	14.81717248
11	2.37	1.185	4.411502944	51.631	11.70372108
12	2.37	1.185	4.411502944	50.234	11.38704896
average				49.0443	11.11736762
std dev				19.71941815	4.469999998

Table 3. Group 2- IPS e.max with 5% hydrofluoric for 20 seconds with Scotchbond

Sample	Diameter	Radius	Surface Area	Peak Load (N)	Peak Stress (MPa)
1	2.37	1.185	4.411502944	82.522	18.70609655
2	2.37	1.185	4.411502944	62.233	14.10698367
3	2.37	1.185	4.411502944	82.286	18.65260004
4	2.37	1.185	4.411502944	37.225	8.438167326
5	2.37	1.185	4.411502944	101.531	23.01505888
7	2.37	1.185	4.411502944	67.74	15.35531107

8	2.37	1.185	4.411502944	32.492	7.365290336
9	2.37	1.185	4.411502944	92.794	21.0345547
10	2.37	1.185	4.411502944	102.18	23.16217427
12	2.37	1.185	4.411502944	97.445	22.08884392
average				75.8448	17.19250808
std dev				25.41907597	5.761999095

Table 4. Group 3- IPS e.max with 5% hydrofluoric for 60 seconds with Scotchbond

Sample	Diameter	Radius	Surface Area	Peak Load (N)	Peak Stress (MPa)
1	2.37	1.185	4.411502944	85.912	19.47454214
2	2.37	1.185	4.411502944	97.132	22.01789305
3	2.37	1.185	4.411502944	104.916	23.78237107
4	2.37	1.185	4.411502944	98.989	22.43883802
5	2.37	1.185	4.411502944	81.649	18.50820481
6	2.37	1.185	4.411502944	86.233	19.54730646
8	2.37	1.185	4.411502944	98.394	22.30396335
9	2.37	1.185	4.411502944	13.228	2.998524577
10	2.37	1.185	4.411502944	71.525	16.21329531
12	2.37	1.185	4.411502944	82.548	18.71199023
average				82.0526	18.5996929
std dev				26.21373965	5.942133549

Table 5. Group 4- IPS e.max with 9.6% hydrofluoric for 20 seconds with Scotchbond

Sample	Diameter	Radius	Surface Area	Peak Load (N)	Peak Stress (MPa)
2	2.37	1.185	4.411502944	66.225	15.0118907
3	2.37	1.185	4.411502944	120.73	27.36709043
4	2.37	1.185	4.411502944	76.775	17.40336592
6	2.37	1.185	4.411502944	71.712	16.25568449
7	2.37	1.185	4.411502944	55.344	12.54538435
8	2.37	1.185	4.411502944	58.491	13.25874668
9	2.37	1.185	4.411502944	98.71	22.37559427
10	2.37	1.185	4.411502944	71.398	16.18450694
11	2.37	1.185	4.411502944	129.114	29.26757652
12	2.37	1.185	4.411502944	102.663	23.27166077
average				85.1162	19.29415011
std dev				26.00519298	5.894860166

Table 6. Group 5- IPS e.max with 9.6% hydrofluoric for 60 seconds with Scotchbond

Sample	Diameter	Radius	Surface Area	Peak Load (N)	Peak Stress (MPa)
1	2.37	1.185	4.411502944	69.234	15.69397117
2	2.37	1.185	4.411502944	74.578	16.90534971

3	2.37	1.185	4.411502944	71.936	16.30646084
4	2.37	1.185	4.411502944	58.68	13.30158922
5	2.37	1.185	4.411502944	116.718	26.4576498
6	2.37	1.185	4.411502944	45.248	10.2568219
7	2.37	1.185	4.411502944	74.41	16.86726745
9	2.37	1.185	4.411502944	59.479	13.48270663
11	2.37	1.185	4.411502944	85.767	19.44167353
12	2.37	1.185	4.411502944	47.483	10.76345196
average				70.3533	15.94769422
std dev				20.63754204	4.678120429

Table 7. Group 6- Empress with Scotchbond

Sample	Diameter	Radius	Surface Area	Peak Load (N)	Peak Stress (MPa)
1	2.37	1.185	4.411502944	72.292	16.38715896
2	2.37	1.185	4.411502944	33.084	7.499484965
4	2.37	1.185	4.411502944	16	3.626881859
5	2.37	1.185	4.411502944	26.01	5.895949823
6	2.37	1.185	4.411502944	25.505	5.781476364
7	2.37	1.185	4.411502944	72.843	16.51205971
9	2.37	1.185	4.411502944	27.051	6.131923824
10	2.37	1.185	4.411502944	32.765	7.427174008
11	2.37	1.185	4.411502944	55.589	12.60092098
12	2.37	1.185	4.411502944	39.603	8.977212642
average				40.0742	9.084024313
std dev				20.0394982	4.542555781

Table 8. Group 7- Empress with 5%hydrofluoric for 20 seconds with Scotchbond

Sample	Diameter	Radius	Surface Area	Peak Load (N)	Peak Stress (MPa)
1	2.37	1.185	4.411502944	9.241	2.094750954
2	2.37	1.185	4.411502944	82.07	18.60363714
3	2.37	1.185	4.411502944	82.997	18.81376961
4	2.37	1.185	4.411502944	102.981	23.34374505
5	2.37	1.185	4.411502944	69.23	15.69306445
6	2.37	1.185	4.411502944	3.857	0.874305208
7	2.37	1.185	4.411502944	63.527	14.40030774
8	2.37	1.185	4.411502944	97.408	22.08045676
11	2.37	1.185	4.411502944	26.956	6.110389213
12	2.37	1.185	4.411502944	80.877	18.33320776
average				61.9144	14.03476339
std dev				35.87001639	8.131019484

Table 9. Group 8- Empress with 5%hydrofluoric for 60 seconds with Scotchbond

Sample	Diameter	Radius	Surface Area	Peak Load (N)	Peak Stress (MPa)
1	2.37	1.185	4.411502944	36.495	8.272690841
3	2.37	1.185	4.411502944	68.204	15.46049065
4	2.37	1.185	4.411502944	31.77	7.201627292
5	2.37	1.185	4.411502944	72.47	16.42750802
6	2.37	1.185	4.411502944	55.49	12.57847965
7	2.37	1.185	4.411502944	76.651	17.37525759
8	2.37	1.185	4.411502944	41.604	9.430799555
9	2.37	1.185	4.411502944	52.928	11.99772519
10	2.37	1.185	4.411502944	57.842	13.11163128
11	2.37	1.185	4.411502944	80.444	18.23505527
average				57.3898	13.00912653
std dev				17.03828696	3.862240867

Table 10. Group 9- Empress with 9.6%hydrofluoric for 20 seconds with Scotchbond

Sample	Diameter	Radius	Surface Area	Peak Load (N)	Peak Stress (MPa)
1	2.37	1.185	4.411502944	63.989	14.50503396
2	2.37	1.185	4.411502944	58.733	13.31360327
4	2.37	1.185	4.411502944	66.545	15.08442833
6	2.37	1.185	4.411502944	33.38	7.566582279
7	2.37	1.185	4.411502944	50.521	11.45210615
8	2.37	1.185	4.411502944	15.704	3.559784545
9	2.37	1.185	4.411502944	20.064	4.548109852
10	2.37	1.185	4.411502944	46.877	10.62608381
11	2.37	1.185	4.411502944	37.124	8.415272634
12	2.37	1.185	4.411502944	31.407	7.11934241
average				42.4344	9.619034723
std dev				17.77461082	4.029150846

Table 11. Group 10- Empress with 9.6%hydrofluoric for 60 seconds with Scotchbond

Sample	Diameter	Radius	Surface Area	Peak Load (N)	Peak Stress (MPa)
2	2.37	1.185	4.411502944	24.536	5.561823331
3	2.37	1.185	4.411502944	58.496	13.25988008
4	2.37	1.185	4.411502944	46.412	10.52067755
6	2.37	1.185	4.411502944	43.39	9.835650242
7	2.37	1.185	4.411502944	60.211	13.64863648
8	2.37	1.185	4.411502944	58.932	13.35871261
9	2.37	1.185	4.411502944	28.623	6.488264966
10	2.37	1.185	4.411502944	68.776	15.59015167

11	2.37	1.185	4.411502944	37.196	8.431593603
12	2.37	1.185	4.411502944	68.97	15.63412761
average				49.5542	11.23295181
std dev				15.96015367	3.617849488

Table 12. Summary

	Stress (MPa)	
Adjusted	Ave	SD
EX-SB	11.11736762	4.469999998
EX-9-20	19.29415011	5.894860166
EX-9-60	15.94769422	4.678120429
EX-5-20	17.19250808	5.761999095
EX-5-60	18.5996929	5.942133549
ES-SB	9.084024313	4.542555781
ES-9-20	9.619034723	4.029150846
ES-9-60	11.23295181	3.617849488
ES-5-20	14.03476339	8.131019484
ES-5-60	13.00912653	3.862240867

Table 13. Charted Summary

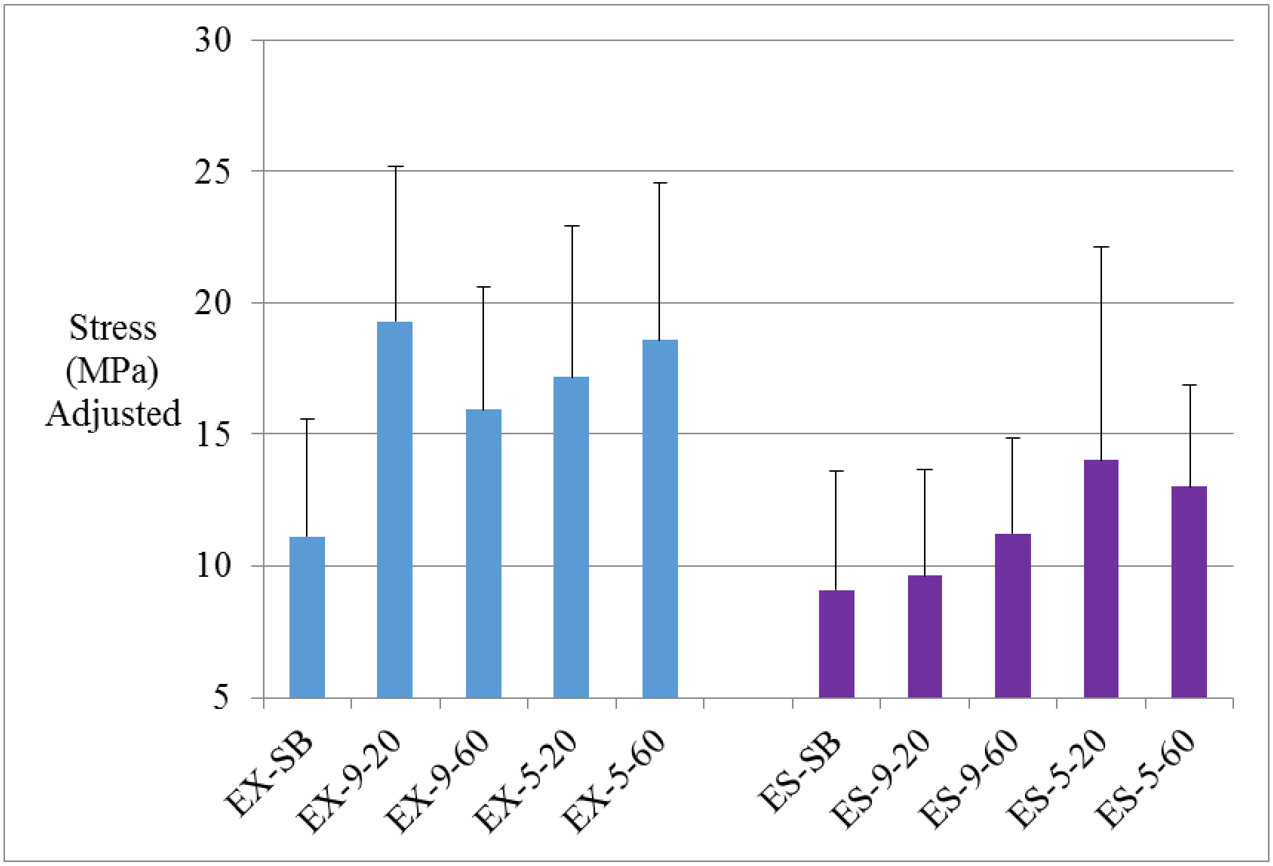


Table 14. Charted Summary with Connecting Letters Report

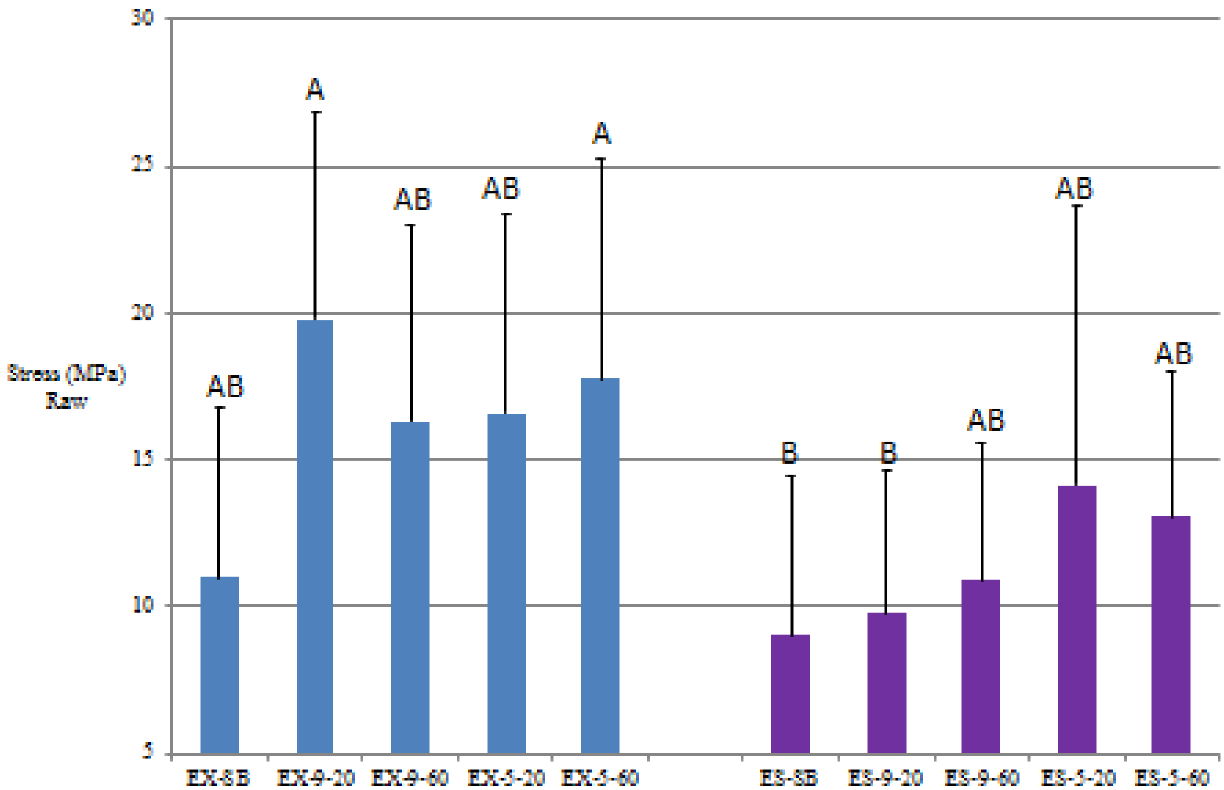
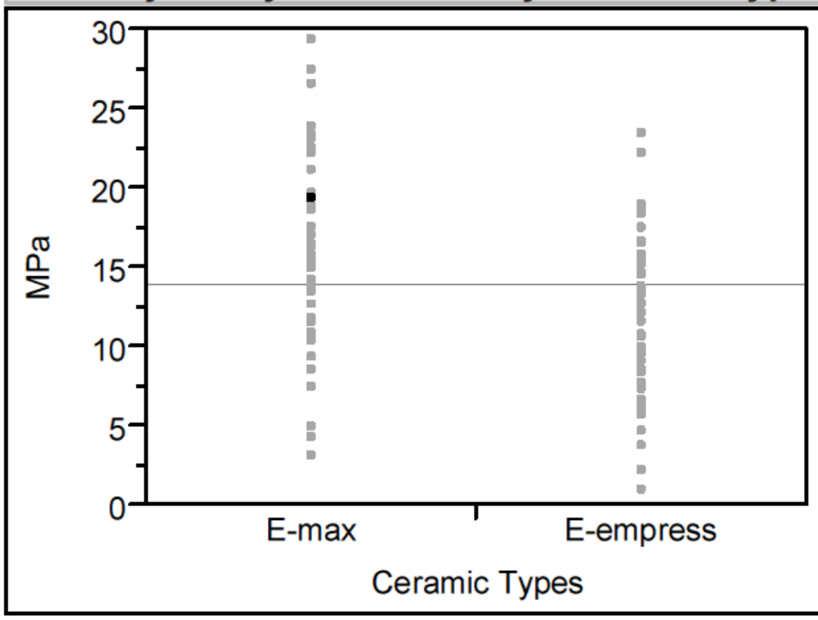


Table 15. Statistical analysis by Ceramic Types

Oneway Analysis of MPa By Ceramic Types



t Test

E-empress-E-max

Assuming unequal variances

Difference	-5.0343	t Ratio	-4.48604
Std Err Dif	1.1222	DF	96.64184
Upper CL Dif	-2.8069	Prob > |t|	<.0001*
Lower CL Dif	-7.2617	Prob > t	1.0000
Confidence	0.95	Prob < t	<.0001*

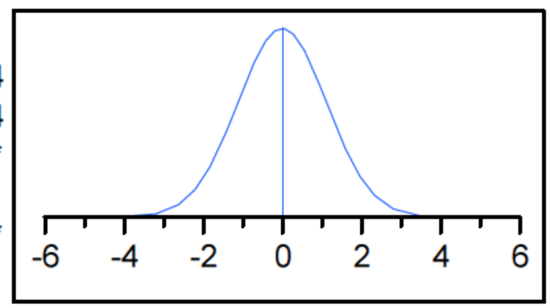
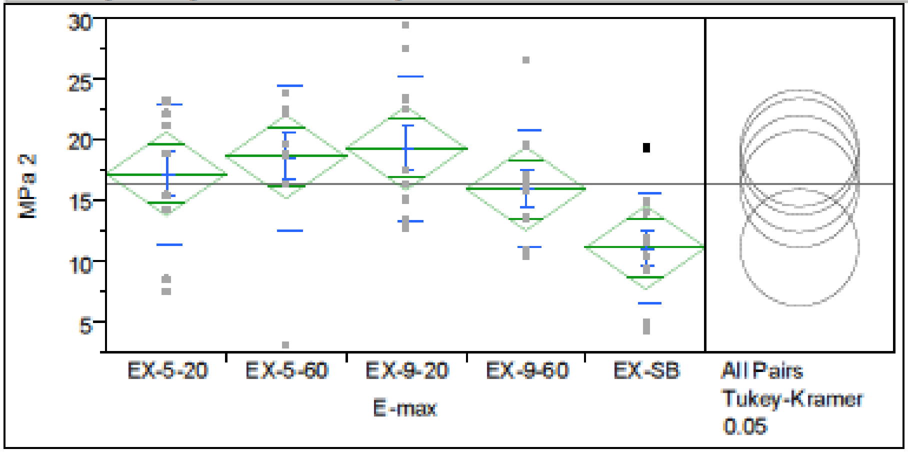


Table 16. Statistical analysis of IPS e.max

Oneway Analysis of MPa 2 By E-max



Missing Rows 50

Oneway Anova

Summary of Fit

Rsquare	0.243096
Adj Rsquare	0.175816
Root Mean Square Error	5.387479
Mean of Response	16.43028
Observations (or Sum Wgts)	50

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
E-max	4	419.4902	104.873	3.6132	0.0123*
Error	45	1306.1220	29.025		
C. Total	49	1725.6123			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
EX-5-20	10	17.1925	1.7037	13.761	20.624
EX-5-60	10	18.5997	1.7037	15.168	22.031
EX-9-20	10	19.2942	1.7037	15.863	22.726
EX-9-60	10	15.9477	1.7037	12.516	19.379
EX-SB	10	11.1174	1.7037	7.686	14.549

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
EX-5-20	10	17.1925	5.76200	1.8221	13.071	21.314
EX-5-60	10	18.5997	5.94213	1.8791	14.349	22.850
EX-9-20	10	19.2942	5.89486	1.8641	15.077	23.511
EX-9-60	10	15.9477	4.67812	1.4794	12.601	19.294
EX-SB	10	11.1174	4.47000	1.4135	7.920	14.315

Means Comparisons

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
2.84145	0.05

Oneway Analysis of MPa 2 By E-max

Means Comparisons

Comparisons for all pairs using Tukey-Kramer HSD

LSD Threshold Matrix

Abs(Dif)-HSD

	EX-9-20	EX-5-60	EX-5-20	EX-9-60	EX-SB
EX-9-20	-6.8461	-6.1516	-4.7444	-3.4996	1.3307
EX-5-60	-6.1516	-6.8461	-5.4389	-4.1941	0.6363
EX-5-20	-4.7444	-5.4389	-6.8461	-5.6013	-0.7709
EX-9-60	-3.4996	-4.1941	-5.6013	-6.8461	-2.0157
EX-SB	1.3307	0.6363	-0.7709	-2.0157	-6.8461

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level			Mean
EX-9-20	A		19.294150
EX-5-60	A		18.599693
EX-5-20	A	B	17.192508
EX-9-60	A	B	15.947694
EX-SB		B	11.117368

Levels not connected by same letter are significantly different.

Ordered Differences Report

Level	- Level	Difference	Std Err Dif	Lower CL	Upper CL	p-Value
EX-9-20	EX-SB	8.176782	2.409354	1.33072	15.02285	0.0120*
EX-5-60	EX-SB	7.482325	2.409354	0.63626	14.32839	0.0259*
EX-5-20	EX-SB	6.075140	2.409354	-0.77092	12.92120	0.1038
EX-9-60	EX-SB	4.830327	2.409354	-2.01574	11.67639	0.2803
EX-9-20	EX-9-60	3.346456	2.409354	-3.49961	10.19252	0.6378
EX-5-60	EX-9-60	2.651999	2.409354	-4.19407	9.49806	0.8052
EX-9-20	EX-5-20	2.101642	2.409354	-4.74442	8.94771	0.9055
EX-5-60	EX-5-20	1.407185	2.409354	-5.43888	8.25325	0.9768
EX-5-20	EX-9-60	1.244814	2.409354	-5.60125	8.09088	0.9853
EX-9-20	EX-5-60	0.694457	2.409354	-6.15161	7.54052	0.9984

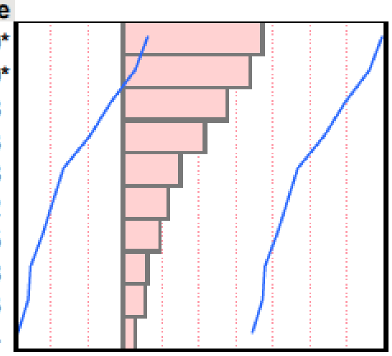
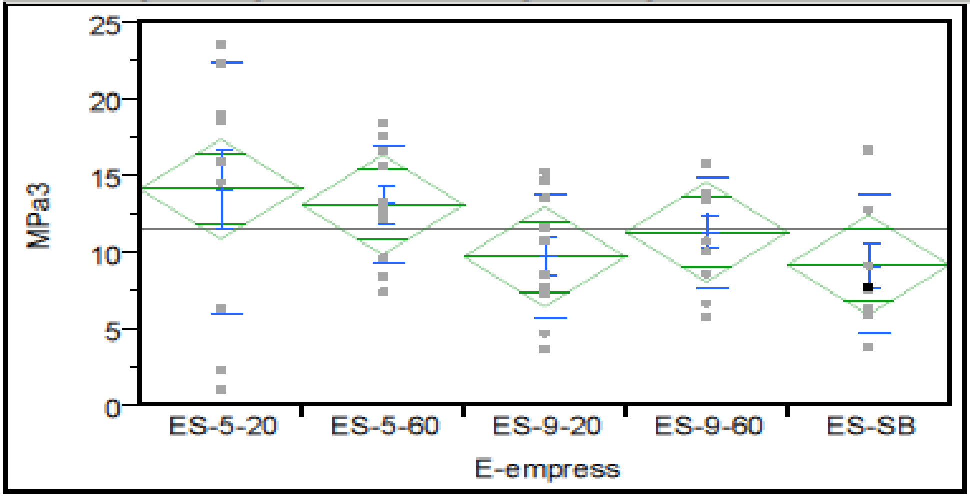


Table 17. Statistical analysis of Empress

Oneway Analysis of MPa3 By E-empress



Missing Rows 50

Oneway Anova

Summary of Fit

Rsquare	0.133065
Adj Rsquare	0.056004
Root Mean Square Error	5.118361
Mean of Response	11.39598
Observations (or Sum Wgts)	50

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
E-empress	4	180.9467	45.2367	1.7267	0.1607
Error	45	1178.8928	26.1976		
C. Total	49	1359.8395			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
ES-5-20	10	14.0348	1.6186	10.775	17.295
ES-5-60	10	13.0091	1.6186	9.749	16.269
ES-9-20	10	9.6190	1.6186	6.359	12.879
ES-9-60	10	11.2330	1.6186	7.973	14.493
ES-SB	10	9.0840	1.6186	5.824	12.344

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
ES-5-20	10	14.0348	8.13102	2.5713	8.218	19.851
ES-5-60	10	13.0091	3.86224	1.2213	10.246	15.772
ES-9-20	10	9.6190	4.02915	1.2741	6.737	12.501
ES-9-60	10	11.2330	3.61785	1.1441	8.645	13.821
ES-SB	10	9.0840	4.54256	1.4365	5.834	12.334

Figure 3. SEM of IPS e.max

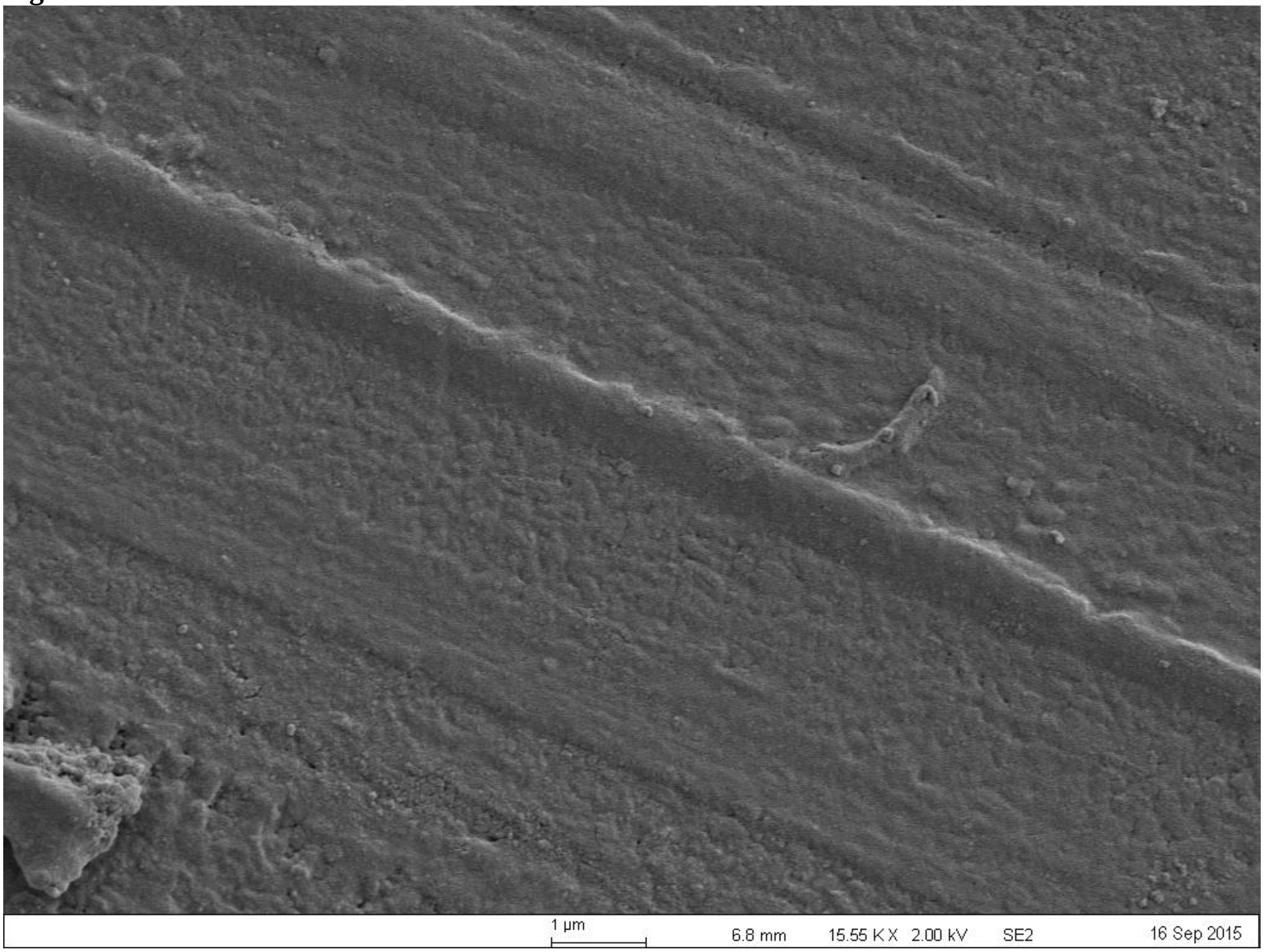


Figure 4. SEM of IPS e.max with 5%HF for 20 seconds

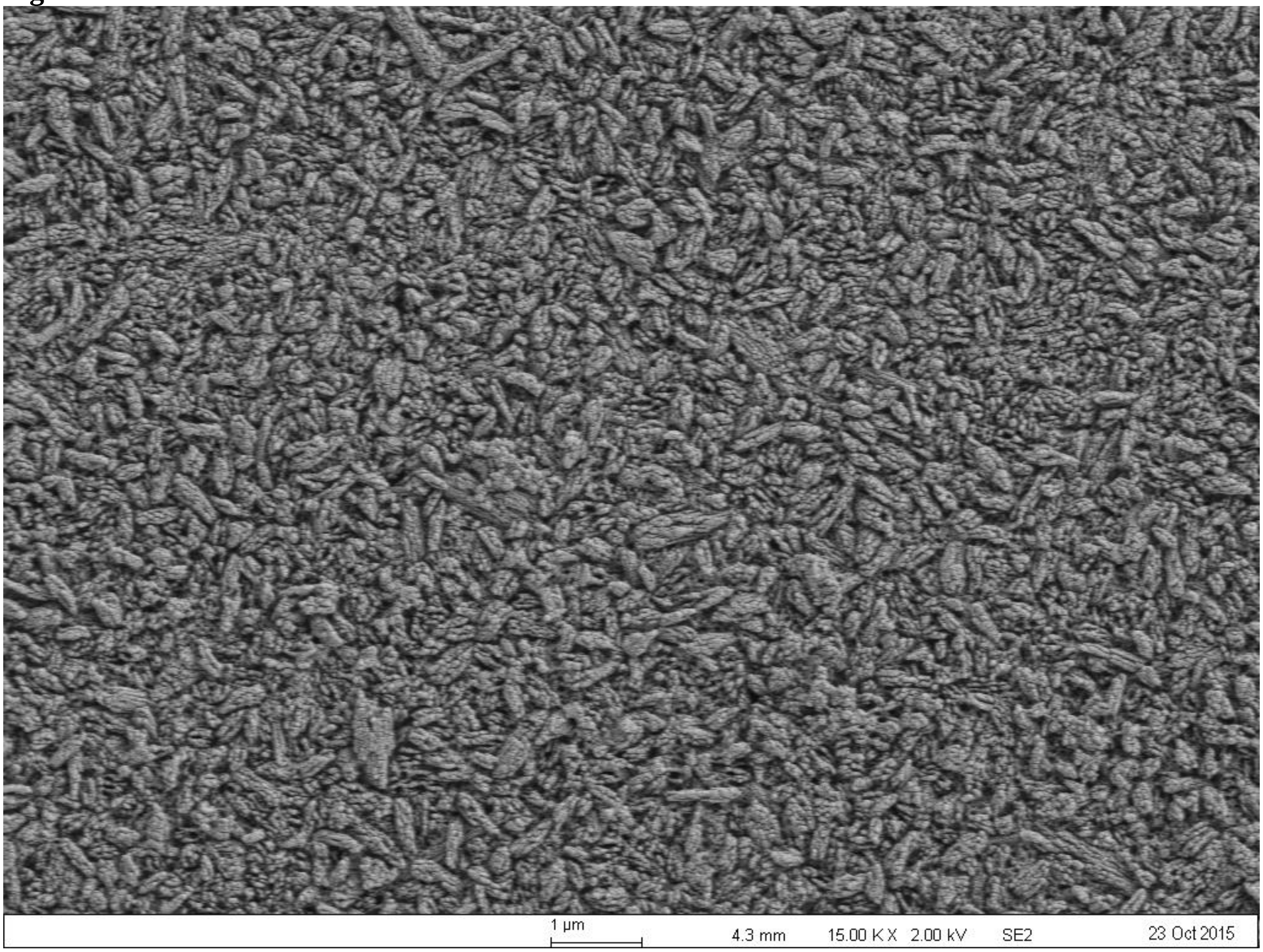


Figure 5. SEM of IPS e.max with 5%HF for 60 seconds

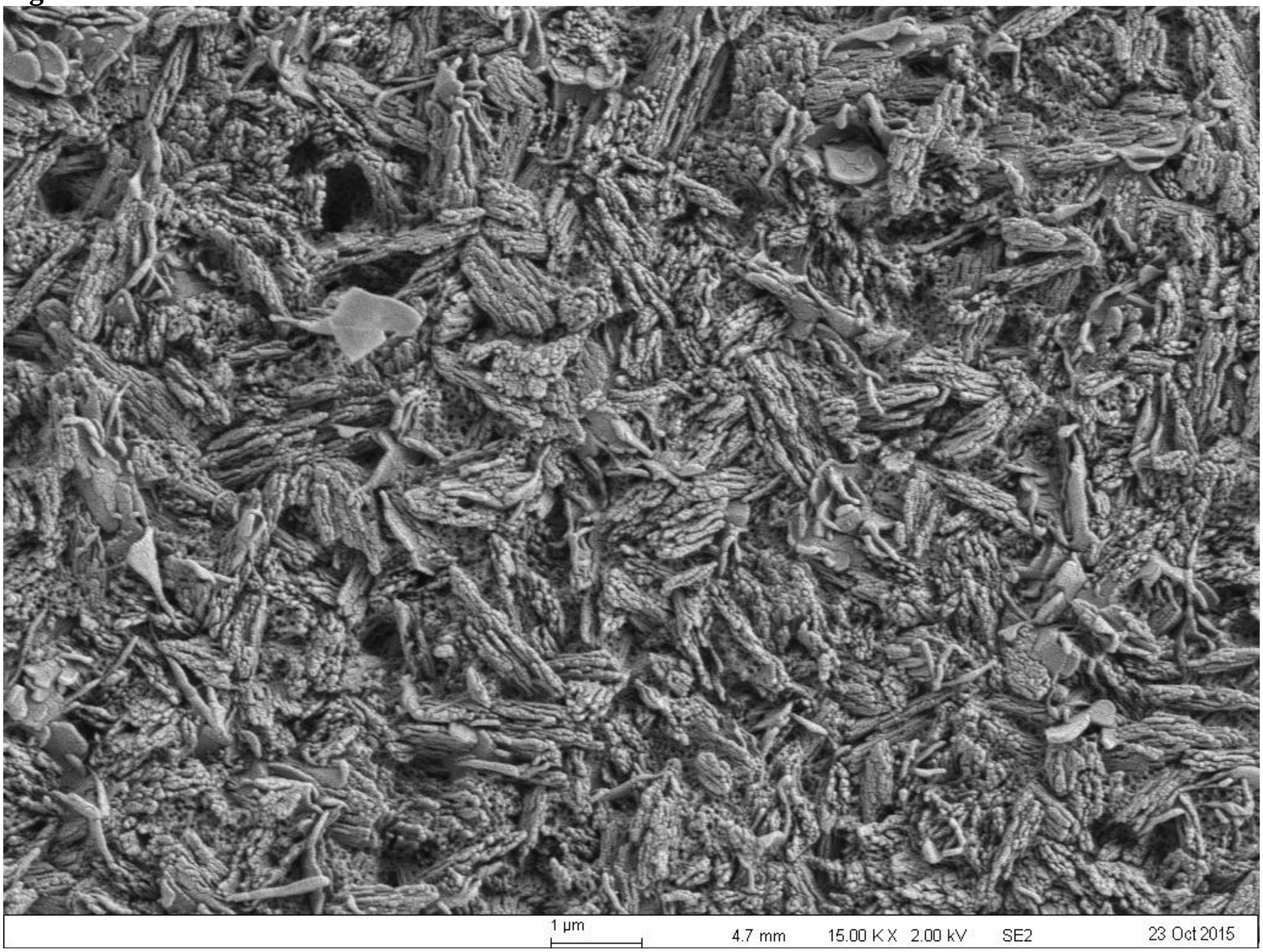


Figure 6. SEM of IPS e.max with 9.6%HF for 20 seconds

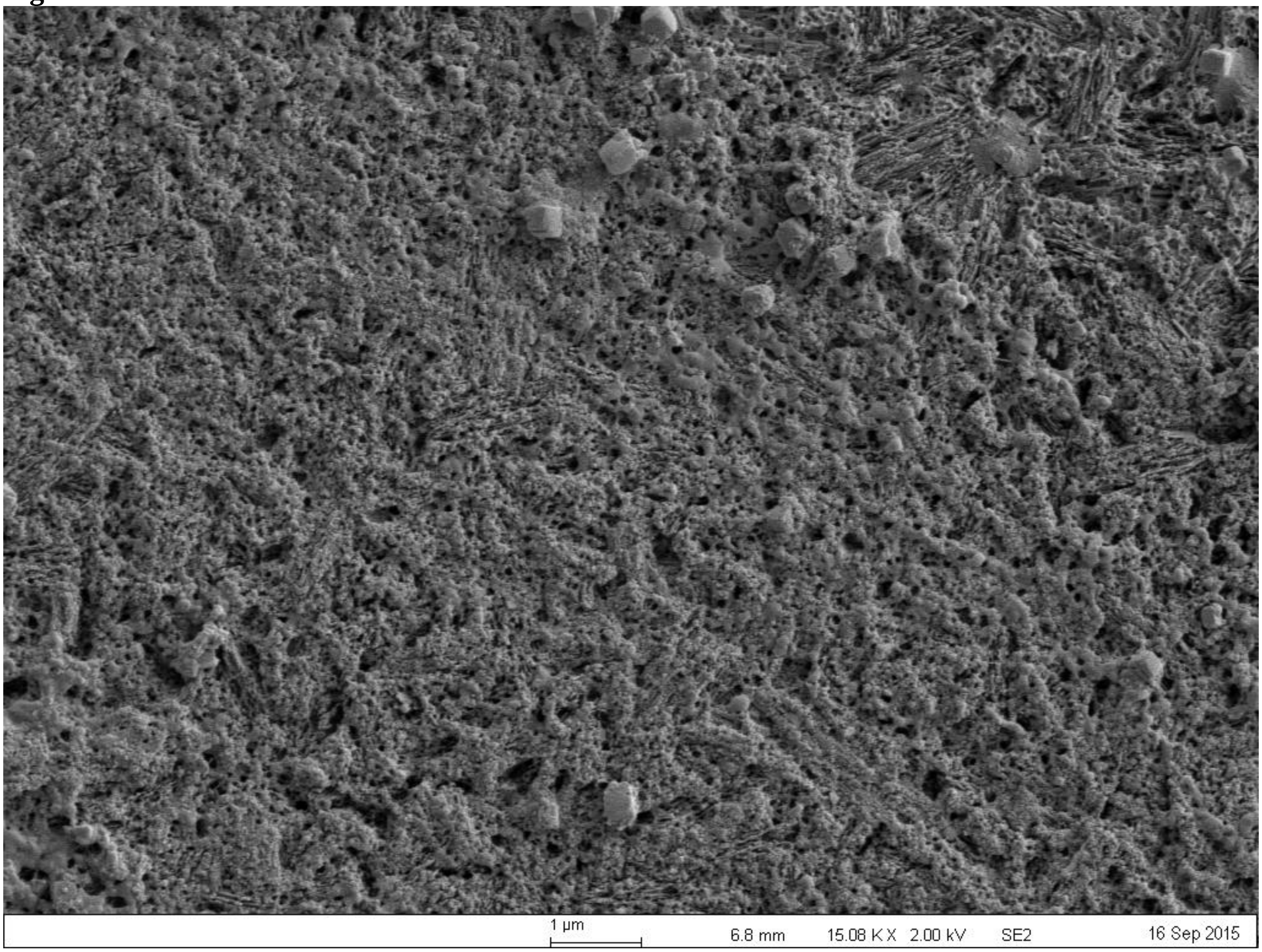


Figure 7. SEM of IPS e.max with 9.6%HF for 60 seconds

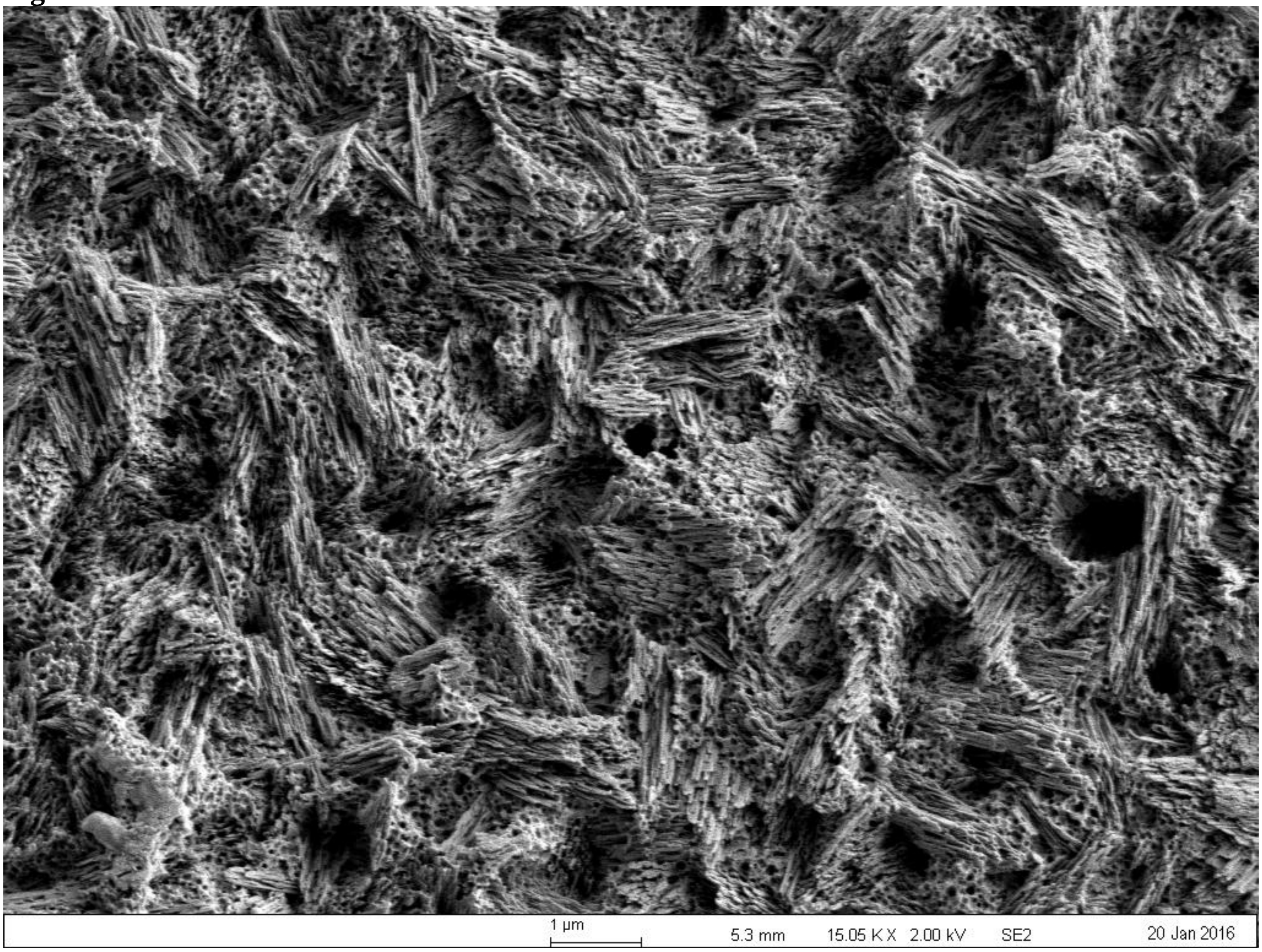


Figure 8. SEM of Empress

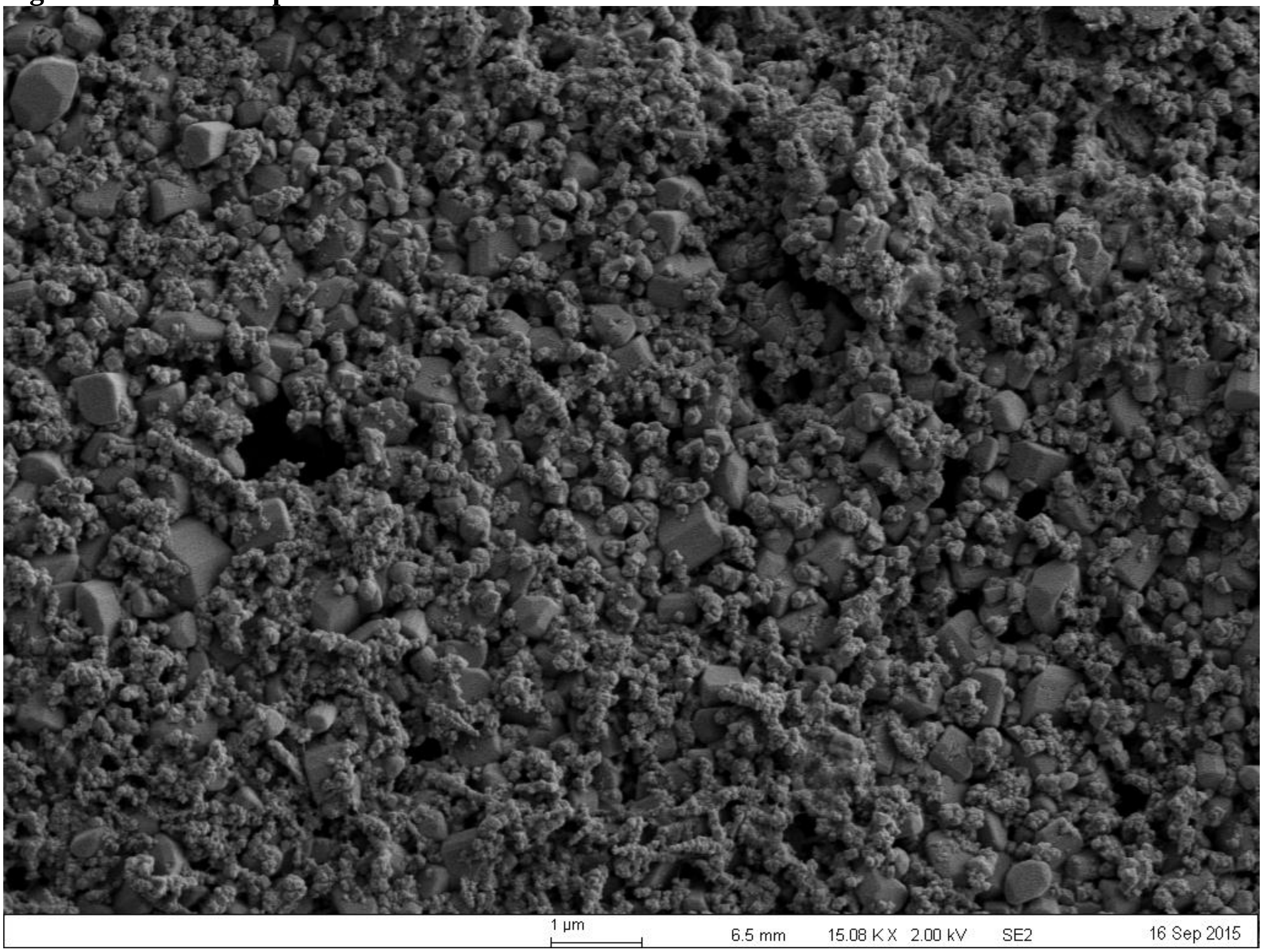


Figure 9. SEM of Empress with 5%HF for 20 seconds

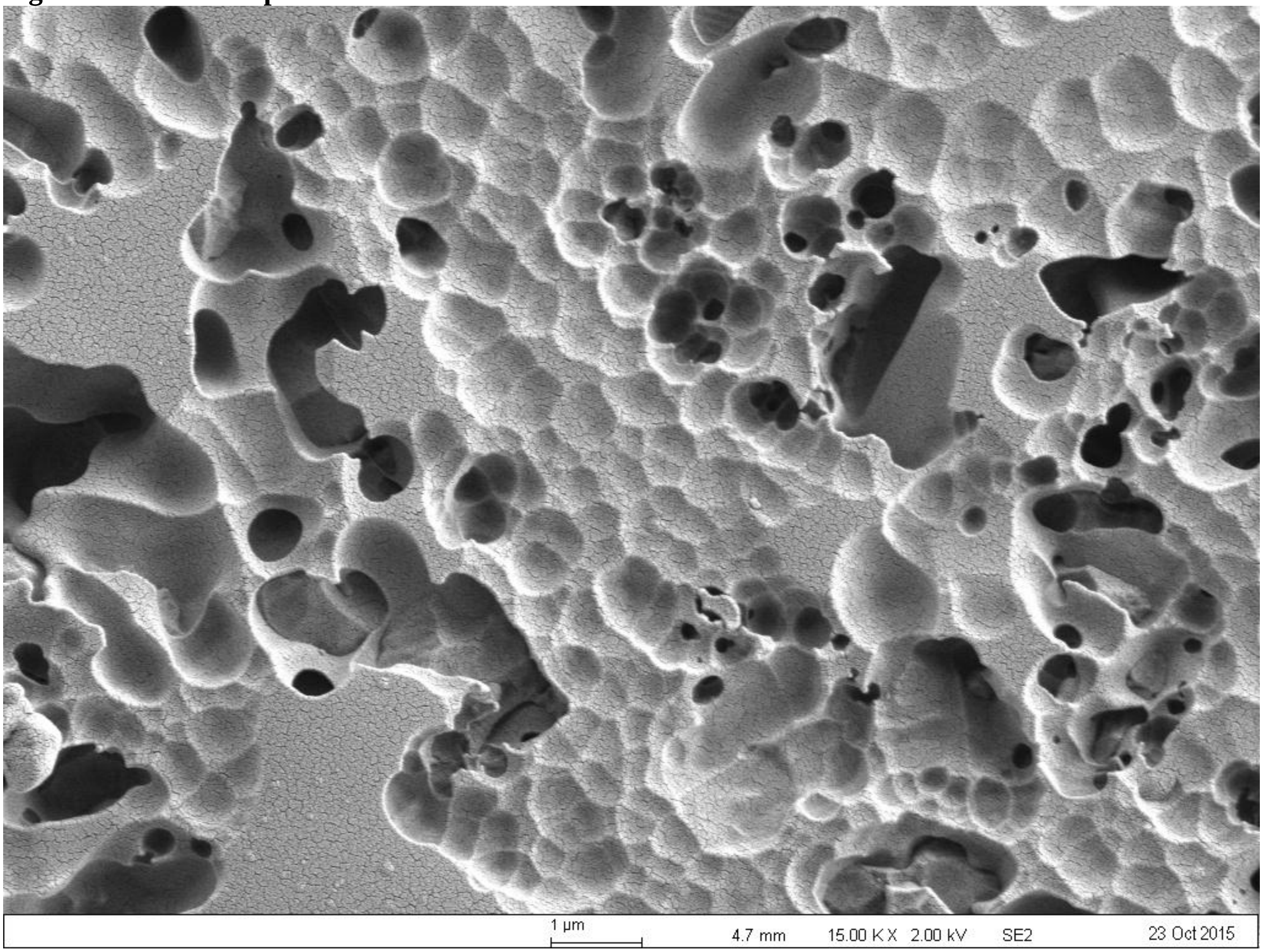


Figure 10. SEM of Empress with 5%HF for 60 seconds

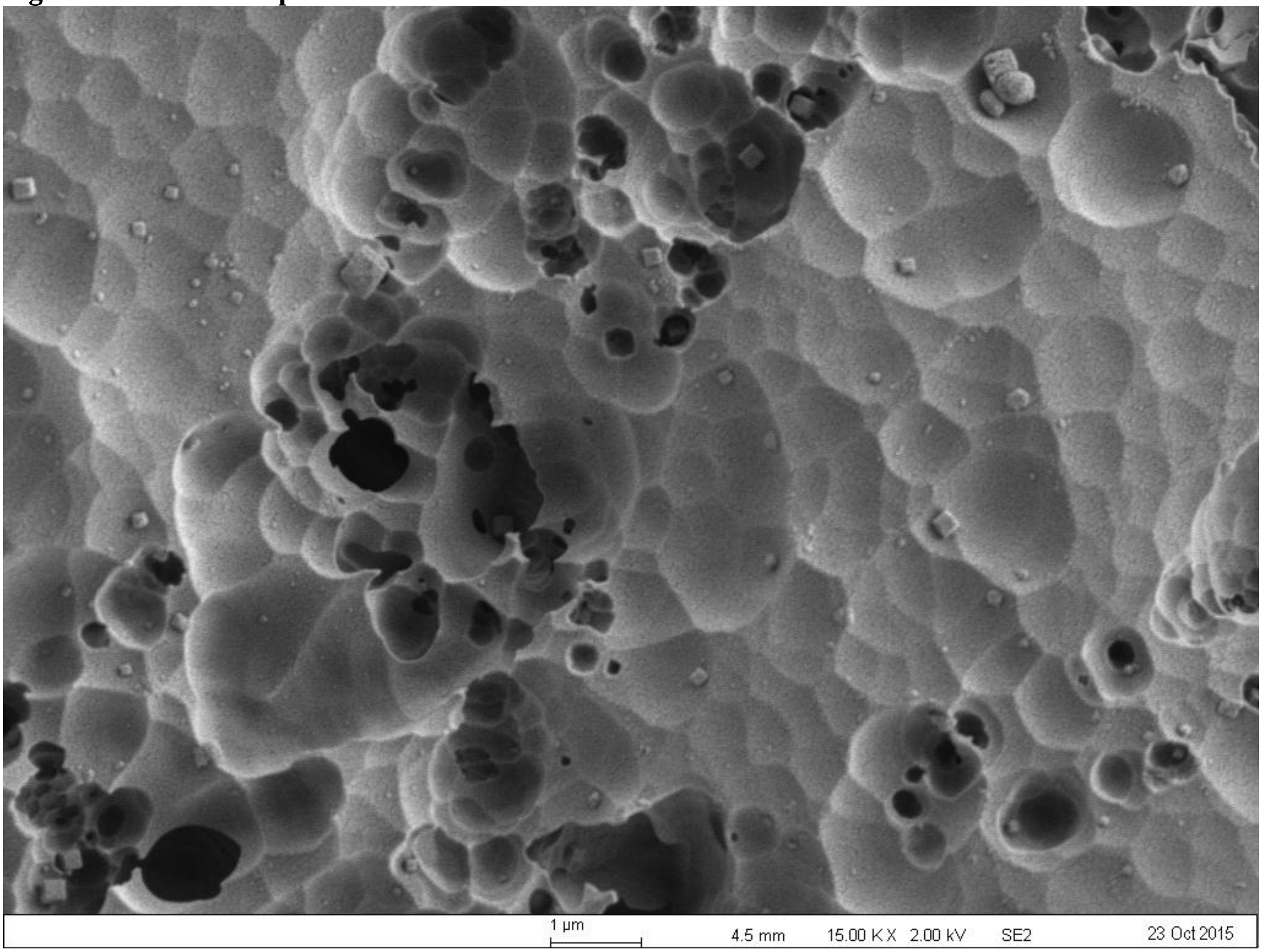


Figure 11. SEM of Empress with 9.6%HF for 20 seconds

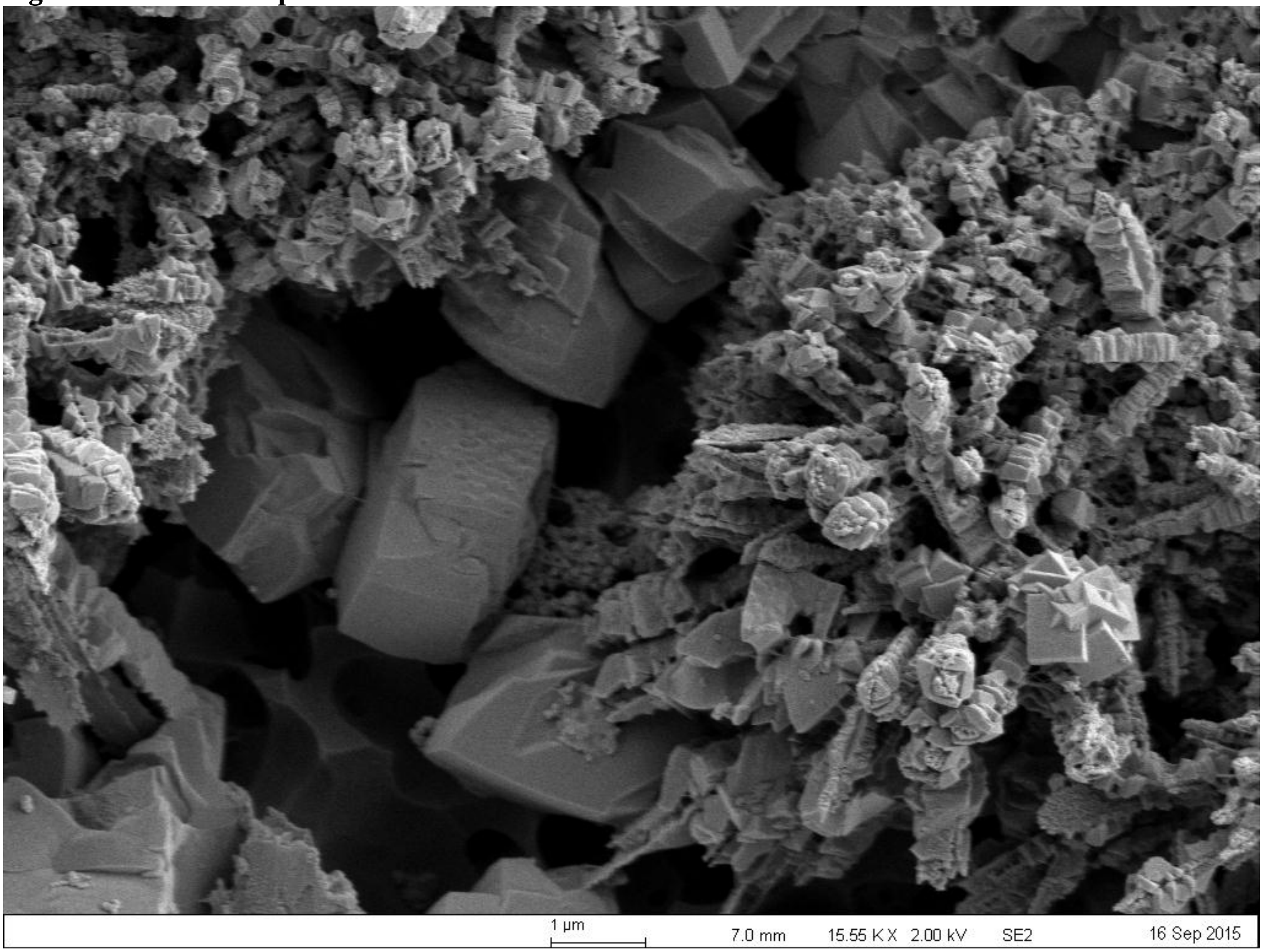
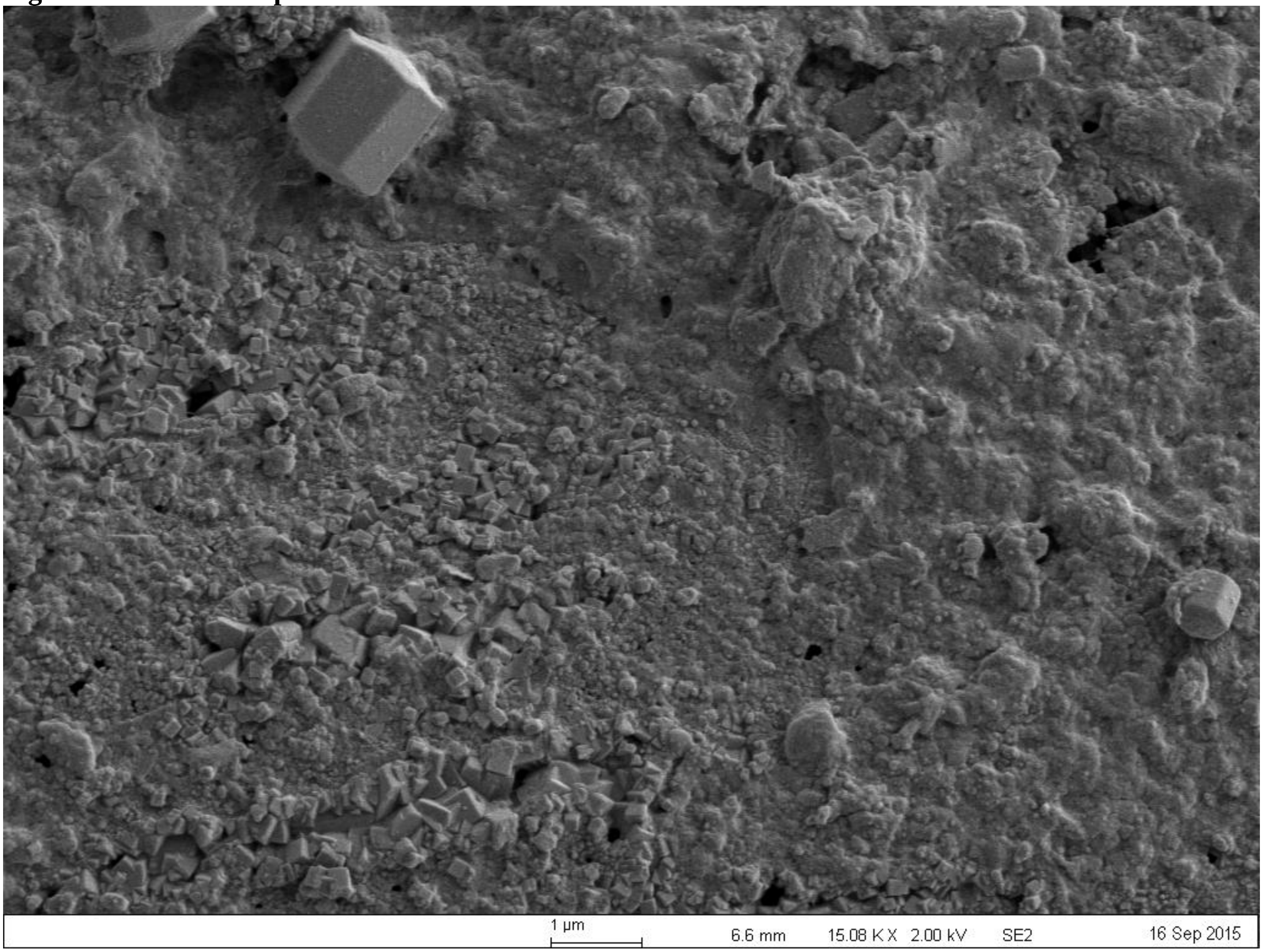


Figure 12. SEM of Empress with 9.6%HF for 60 seconds



DISCUSSION

In this study, the shear bond strength of two ceramic materials was compared with differing surface preparations. In designing this study an International Standard is available to provide a standard process for determining shear bond strength. For preparation and performing this study ISO 29022, Dentistry-Adhesion-Notched-edge shear bond strength test, was followed. For the study a knife edge shear blade or notched edge shear blade could be used. The knife edge blade would provide shear force at a minimal area of the composite button with the notched edge contacting an increased area of composite button. To spread the force over a greater area of bonded composite structure the notched edge shear blade was chosen. One of the limitations of this study is the use of static loading until fracture which is less clinically significant than of applying cyclic loading. This in-vitro study does not show or demonstrate how the bond strength is affected by continual or cyclic loading, but shows the highest initial bond strength of composite buttons to ceramic materials with different surface preparations.

In a 2003 literature review by Blatz et al, it was reported that the high content of lithium disilicate crystalline phase in IPS e.max may produce higher adhesion values than Empress or similar materials with leucite or feldspathic ceramics, regardless of the surface treatment that the materials were subjected to. [20] This literature review does explain the similar results obtained in this study between IPS e.max and Empress with IPS e.max having the higher shear bond strength.

Chemically IPS e.max and Empress are very similar. [21] The ability to adhere the composite resin to the porcelain in IPS e.max and Empress is due to the chemical coupling agent, silane. [21] Scotchbond Universal, the silane coupling agent used for the study, was used to surface treat the glass particles in the porcelain. [21] Scotchbond Universal has the ability to form a durable bond between organic and inorganic materials. [22] The adhesive mechanism is thought to be due to the low molecular weight and low surface energy of the silanes allowing them initially to spread into thin films and penetrate porous structures. [22] The porous structure of the porcelain crowns after etching allows crosslinking which results in the formation of a silica-rich encapsulating network. [22]

In this study all fractures were adhesive failures. The failure occurred at the weakest part which was the chemical and mechanical bond between the composite and ceramic. Comparing IPS e.max to Empress in Table 14 it shows IPS e.max has consistently higher bond strengths in all surface preparations. Table 15 shows the t-test analysis confirming a statistically significant difference between the bond strengths of IPS e.max compared to Empress.

Comparing IPS e.max Table 2 to Empress Table 6 with Scotchbond treatment only the IPS e.max has 2 MPa more bond strength. Analyzing the SEM images Figure 3 for IPS e.max and Figure 8 for Empress the IPS e.max is a smooth surface compared to the higher surface area of Empress. The higher bond strength of e.max could be explained by the literature review by Blatz et al. My search of the literature was unable to find any articles to explain why IPS e.max produces higher shear bond strength than Empress regardless of the surface treatment.

Comparing IPS e.max Table 3 to Empress Table 7 with 5% hydrofluoric acid for 20 seconds with Scotchbond showed IPS e.max with 17.19 Mpa average and Empress with 14.03 Mpa average. Figure 4 shows the 5% hydrofluoric acid was able to etch the glass and greatly improve the surface area of IPS e.max. This surface area provided the increase in shear bond strength compared to the unetched IPS e.max. Figure 9 shows that when Empress is etched with 5% hydrofluoric acid no glass is etched, but the Empress seems to be dried out with fissures on its surface. The fissures and rounded surfaces of Empress seen in the SEM could explain the increase in surface area to Scotchbond and composite which allows the increase in shear bond strength compared to unetched Empress.

When applying 5% hydrofluoric acid for 60 seconds to IPS e.max, Table 4, the shear bond strength increased to 18.6 Mpa. Figure 5 displays the glass is etched more and the configuration of IPS e.max crystals is disorderly. The disorder does create an increased amount of surface area compared to Figure 4 IPS e.max that was etched for 20 seconds with 5% hydrofluoric acid. Figure 10 shows Empress etched for 60 seconds compared to 20 seconds in Figure 9 with 5% hydrofluoric acid differs. When Empress is etched for 60 seconds it does display the cracks and fissures as 20 seconds displays. When etched for 60 seconds Empress does still display rounded surfaces and some glass has been plucked from the Empress. The lack of fissures creating a decreased surface area explains the decrease in shear bond strength to 13.0 Mpa, Table 9.

Changing to a different concentration of hydrofluoric acid, 5% to 9.6%, provided a slight change in shear bond strength for IPS e.max and a greater change in Empress shear bond strength. With etching for 20 seconds with 9.6% hydrofluoric acid provided the highest shear bond strength of the experiment with IPS e.max at 19.29 Mpa average, Table 5 . Figure 6 displays the SEM of IPS e.max from the group with a large increase in surface area and porosities. This large amount of surface area compared to the other groups of etched and non-etched IPS e.max displays why this group has the highest shear bond strength. Figure 11 displays Empress when etched with a large amount of glass removed. The leucite crystals can be visualized and large voids in between larger crystals. In my study, I was not able to determine the exact surface area to compare the etched E.max and Empress. In this group, a determination of whether E.max or Empress had a greater surface area would help in the correlation to bond strength. It could be extrapolated that this vast amount of surface area would provide Empress with higher shear bond strength. After testing, Empress showed the second lowest shear bond strength in this category at 9.61 Mpa ,Table 10,and only stronger than Empress to Scotchbond. From the data, the smooth rounded surfaces and fissures of Empress provide a greater bond strength. My search of the literature was unable to find any articles to explain the greater shear bond strength of smooth and rounded Empress topography.

The last group was exposed to the 9.6% hydrofluoric acid for 60 seconds prior to Scotchbond and composite application, IPS e.max Table 6, Empress Table 11. IPS e.max shear bond strength decreased in comparison to etching for 20 seconds to 15.9 Mpa. The SEM image of e.max, Figure 7, displays more linear crystal projections and higher crystal ridges compared to etching for 20 seconds. The depth of glass dissolution is less in the 60 second etch group compared to 20 seconds. Empress shear bond strength at 11.2 Mpa was in the middle for the different surface treatments of Empress. The SEM of Empress shows the glass etched and leucite crystals visible. This increased surface area increased the shear bond strength compared to etching for 60 seconds.

Performing a 1 way ANOVA for IPS e.max as shown in Table 15 displays the probability is 0.0123 showing there is statistically significant differences between the surface preparations. A Tukey-Kramer post hoc test was conducted displaying the connecting letters report on Table 14. The report shows that IPS e.max prepared with 9.6% hydrofluoric acid for 20 seconds and IPS e.max prepared with 5% hydrofluoric acid for 60 seconds are significantly different than the other surface preparations.

For Empress analysis, Table 16, 1 way ANOVA calculates probability of 0.1607, determining there is no statistically significant difference in the shear bond strength for surface preparations between Empress. Due to the ANOVA probability greater than 0.05 a Tukey-Kramer post hoc test was not conducted.

Based on the results of this study, to place composite buttons on IPS e.max crowns the surface treatment to provide the highest shear bond strength is 9.6% hydrofluoric acid for 20 seconds. The higher shear bond strength will decrease the number of additional appointments due to debonding of brackets and composite buttons. The higher shear bond strength will increase the life span of fractured porcelain crowns repaired with composite. This surface treatment will decrease chair time of patient, dentist, staff and additional materials for rebonding and repairing. This study was unable to find a surface treatment of Empress that statistically provided the highest shear bond strength.

CONCLUSION

Based on the results of this in-vitro study, one can conclude that IPS e.max® CAD etched with 9.6% hydrofluoric acid for 20 seconds showed the highest shear bond strength of all materials tested with IPS e.max® CAD etched with hydrofluoric 5% acid for 60 seconds also statistically significant.

Null hypothesis: There was no difference in shear bond strength between lithium disilicate and leucite reinforced feldspathic restorations regardless of the surface preparation used.

Based on the conclusion, the null hypothesis was rejected due to IPS e.max® CAD etched with 9.6% hydrofluoric acid for 20 seconds showing significantly higher shear bond strength than other surface preparations.

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